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(54) Phase change thermal interface material

(57) A thermal interface material (A) transfers heat from a heat source (12), such as a microprocessor, to a heat sink (14) for maintaining the microprocessor at a safe operating temperature. The interface material includes thermally conductive filler particles dispersed in a phase change material. The phase change material softens and flows at the operating temperature of the heat source, thereby providing good thermal contact with uneven surfaces of the heat source and heat sink, without excessive exudation and loss of the interface material. The phase change material includes a polymer component, such as an elastomer, and a melting point component, which adjusts the softening temperature of the phase change material to the operating temperature of the heat source.

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**Description****Background of the Invention**

- 5 [0001] This invention pertains to thermally conductive materials, and more particularly, to a thermally conductive interface material for heat generating devices, such as microprocessor power supply assemblies, that facilitates heat transfer from the heat generating device to a heat sink.
- [0002] With increasing market pressure for smaller, faster, and more sophisticated end products using integrated circuits, the electronics industry has responded by developing integrated circuits which occupy less volume, yet operate  
10 at high current densities. Power supply assemblies for such microprocessors generate considerable heat during operation. If the heat is not adequately removed, the increased temperatures generated by the power supply assemblies will result in damage to the semiconductor components.
- [0003] A heat sink is commonly used to transfer the heat away from the power supply or other heat generating assembly. The heat sink generally includes a plate or body formed from a conductive metal, which is maintained in thermal contact with the assembly for dissipating heat in an efficient manner. Fins optionally protrude from the plate for providing an increased surface area for heat dissipation to the surrounding environment.  
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- [0004] The current industry technique for providing thermal contact between a microprocessor power supply assembly and a heat sink is to interpose a thermal interface material between the two, which facilitates heat transfer from the active device to the heat sink.
- 20 [0005] One method is to apply a ceramic filled thermal grease, which is typically silicone based, between the heat sink and the power supply. Thermal greases provide excellent thermal properties, but require an extensive assembly process with high manufacturing cost. The product is usually applied by hand, from a syringe, or with an aluminum carrier. This process is labor intensive and slow and does not lend itself to automation.
- [0006] Another method for providing a conductive interface includes the use of thermally conductive wax compounds.  
25 These compounds, however, are generally brittle at ambient temperatures and easily chipped off, resulting in a high thermal resistance. The low viscosity of the wax at operating temperature causes the wax to flow out from between the active component and the heat sink, resulting in a high thermal resistance. Further, because of the brittle nature of the wax compounds, they are difficult to manufacture and apply to a heat sink.
- [0007] Thermally conductive silicone rubbers have also been used as conductive interfaces. Although soft and pliable,  
30 the silicone rubber requires relatively high pressure and a long warm-up time to attain a low thermal resistance. The rubbers have poor flow characteristics which result in a low thermal conduction when there is a mismatch of flatness between the heat sink and the heat producing device. Differences in the thermal coefficient of expansion between the silicone rubber and the heat sink can result in high thermal resistance during temperature cycling. These effects lead to a poor thermal conductivity from the heat producing device to the heat sink.
- 35 [0008] Other thermal interfaces employ polymeric thermally conductive cure-in-place compounds. These compounds are generally rigid after cure. They have a poor reliability because of a difference in thermal coefficient of expansion between the material and the heat sink, causing cracks and failure during temperature cycling. The polymeric materials are labor intensive to apply and require long cure times.
- [0009] The present invention seeks to provide a new and improved thermal interface which overcomes the above-  
40 referenced problems and others.

**Summary of the Invention**

- 45 [0010] The present invention relates to a thermal interface material which can be easily pre-attached to a microprocessor power assembly or a heat sink prior to shipment.
- [0011] In accordance with one aspect of the present invention, a thermal interface material, which undergoes a phase change at microprocessor operating temperatures to transfer heat generated by a heat source to a heat sink, is provided. The thermal interface material includes a phase change substance, which softens at about the operating temperature of the heat source. The phase change substance includes a polymer component and a melting point component.  
50 The melting point component modifies the temperature at which the phase change substance softens. The thermal interface material further includes a thermally conductive filler dispersed within the phase change substance.
- [0012] In accordance with another aspect of the present invention, a multi-layer strip is provided. The strip includes a layer of a thermal interface material for thermally connecting a heat source with a heat sink. The thermal interface material includes a polymer component, a melting point component in sufficient quantity to adjust the softening temperature of the interface material to about the operating temperature of the heat source, and a thermally conductive filler mixed with the melting point component and the polymer component. The strip further includes an outer layer disposed on a side of the thermal interface material. The outer layer includes at least one of a protective releasable liner and a layer of an adhesive material.  
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- [0013] In accordance with another aspect of the present invention, a method of providing a thermal interface between a heat source and a heat sink is provided. The method includes interposing a thermal interface material between the heat source and heat sink, which softens at about the operating temperature of the heat source to provide a thermal interface between the heat source and the heat sink during operation of the heat source. The thermal interface material includes a polymer component, a melting component for modifying the temperature at which the thermal interface material softens, and a thermally conductive filler mixed with the polymer component and the melting point component.
- [0014] One advantage of the present invention is that the thermal interface material can be pre-attached to a heat sink prior to shipment.
- [0015] Another advantage of the present invention is that the cohesive strength and integrity of the thermal interface material provide for easy handling.
- [0016] Still another advantage of the present invention is that the thermal performance of the thermal interface material matches that of thermal grease in a solid film form.
- [0017] Still another advantage of the present invention is that a phase change or softening at the operating temperatures maximizes interfacial surface wetting.
- [0018] Still another advantage of the present invention is that low application pressure without added heat allows for hand mounting during field rework and processor upgrades.
- [0019] Still another advantage of the present invention is that the assembly process associated with thermal grease is eliminated but an equivalent thermal performance is maintained.
- [0020] Still another advantage of the present invention system assembly cost is minimized by allowing for pre-attachment to a heat sink or CPU.
- [0021] Still another advantage of the present invention is that the material softens and conforms to surface roughness or concavity at operating temperature.
- [0022] Still another advantage of the present invention is that the material operates at low clip pressures (5 to 10 psi).
- [0023] Still another advantage of the present invention is that the material can be applied and repositioned with thumb pressure allowing for easy field service.
- [0024] Still another advantage of the present invention is that the material allows for vertical mounting due to its cohesive properties.
- [0025] Still other benefits and advantages of the invention will become apparent to those skilled in the art upon a reading and understanding of the following detailed specification.
- [0026] As used herein with respect to the thermally conductive filler and the polymer component the terms "mixed" and "dispersed" are used interchangeably.

### 35 Brief Description of the Figures

- [0027] The invention may take form in various components and arrangements of components, and in various steps and arrangements of steps. The drawings are only for purposes of illustrating a preferred embodiment and are not to be construed as limiting the invention.
- [0028] FIGURE 1 is a schematic view of a heat sink, heat source, and intermediate thermally conducting film prior to assembly, according to the present invention;
- [0029] FIGURE 2 is a side sectional view of a first embodiment of a multi-layer strip comprising the film of FIGURE 1;
- [0030] FIGURE 3 is a side sectional view of a second embodiment of a multi-layer strip;
- [0031] FIGURE 4 is a side sectional view of a further embodiment of a multi-layer strip;
- [0032] FIGURE 5 is a schematic side sectional view of a heat sink with the multi-layer strip of FIGURE 2 attached;
- [0033] FIGURE 6 is a side sectional view of the heat sink, heat source and attached thermally conducting film of FIGURE 1; and
- [0034] FIGURE 7 is a plot of thermal impedance versus mounting pressure for three interface materials.

### 50 Description of the Preferred Embodiment

- [0035] With reference to FIGURE 1, a thermally conductive, interface material A in the form of a film or layer 10 provides a thermal interface between an active device or heat source 12, such as a microprocessor power supply assembly, and a heat sink 14, such as a block of heat transmissive material, to facilitate heat transfer from the device 12 to the heat sink 14. It will be appreciated that the heat source or device 12 can be any type of semiconductor device or power supply assembly that generates excess heat in its operation, which heat if not removed, may damage the device or impair operation of the device.

- [0029] The film 10 is preferably of from about 0.025 to 2.5 millimeters in thickness. The film thickness can be further increased, if desired, to accommodate certain application requirements, such as larger spacing characteristics in electronics or power supply cooling application.
- [0030] The interface material A consists of a mixture of a phase change substance and a thermally conductive filler. 5 The interface material A has a sufficient cohesive strength to allow handling in a roll form or as pre-cut pieces. The design of the product allows the use of die-cut parts mounted to a bandoleer web to supply continuous parts to a manual or automated part dispensing or "pick and place" part application process.
- [0031] With reference also to FIGURES 2-4, the material A is preferably supplied in the form of a multi-layer strip 10 in which the film 10 of the interface material is sandwiched between layers of adhesive and/or releasable protective liners. FIGURE 2 shows a first embodiment of a multi-layer strip 20 in which a protective liner 22 is supplied on one or more sides of the film 10 of thermally conductive material A. The protective liner preferably comprises a coated substrate, such as SCK polyethylene-coated paper (e.g., PN 907826, 20", or 909785, 24"), polyethylene or polyester film, coated with a release coating, such as a polydimethyl siloxane, fluorosilicone, or non-silicone release coating. Examples of such laminates include blue poly 2.5 mil 2S PN 9099037. One or both sides of the liner substrate may be coated 15 with release coating as desired. The protective liner protects the film of thermally conductive material against damage prior to application to the heat sink or active device. The liner 22 is peeled off prior to use.
- [0032] With reference to FIGURE 3, a second embodiment of a multi-layer strip 30 includes first and second liner layers 22 and 34 which form the outermost layers of the strip 30. Intermediate between the film 10 of thermally conductive material and one or more of the protective layers 34 is a layer of adhesive 36 to aid in attachment of the film to the 20 heat sink 14 or power supply assembly 12. The adhesive is preferably a pressure sensitive adhesive, which permits the thermally conductive film to be attached to a heat sink or to a power supply with minimal pressure, typically less than 10 psi, and without the need for heat. Where the adhesive is not employed, the thermally conductive film 10 is applied to the heat sink or to the power supply by providing a small amount of heat to the layer.
- [0033] With reference to FIGURE 4, a third embodiment of a multi-layer strip 40 includes a reinforcement layer 42 25 between the thermally conductive film 10 and adhesive layer 36. The reinforcement layer is provided to improve the mechanical strength of the film. The reinforcement layer may include a polymeric film, woven or non-woven cloth, such as glass cloth (formed from fiberglass), or a metallic film, such as aluminum. Alternatively, reinforcement materials, such as fibers or woven cloth, may be embedded in the thermally conductive film 10 itself.
- [0034] It should be readily appreciated that other configurations of the multi-layer strip are also possible depending 30 on whether the film 10 is to be attached to one or the other or both of the heat sink 14 and the microprocessor 12 prior to their shipment, and whether the attachment is to take place in the field, where heat for joining the film to the two parts, 12, 14, is not readily available, and thus an adhesive layer, or layers, is desired. For example, if the strip is to be attached to both parts 12, 14, in the field, the strip may include two layers of adhesive, one for attaching the film to the heat sink, the other for attaching the film to the power assembly.
- [0035] With reference to FIGURE 5, a multi-layer strip 20 of the type illustrated in FIGURE 2 is shown mounted to 35 a heat sink (e.g., by application of pressure and/or heat). The protective layer 22 remains in place until it is desired to attach the heat sink to a microprocessor 12 (or to a power transistor or other heat-generating device).
- [0036] With reference now to FIGURE 6, the protective layer 22 has been removed from the multi-layer strip, and the exposed side of the thermally conductive film 10 has been attached to the microprocessor 12.
- [0037] During use, the heat generated by the microprocessor warms the thermally conductive film 10. The film 40 undergoes a softening or phase change at or just below the operating temperature of the microprocessor, allowing wet-out of the heat sink and microprocessor surfaces by the thermally conductive material A. This results in the elimination of thermally insulating air pockets. After initial wetting, the material A proceeds to transfer the heat generated by the microprocessor 12 to the heat sink 14.
- [0038] The thermal performance of the thermally conductive film 10 matches that of ceramic filled greases commonly used in the industry. However, the film retains a relatively high melt viscosity to eliminate excessive flow and dripping from vertically mounted assemblies. During thermal cycling (on/off switching of the heat generating device 12) the film 10 maintains interfacial contact and excellent thermal performance.
- [0039] The specific formulation of the film 10 is preferably selected according to the conditions to which the film is 50 to be exposed (e.g., operating temperature, temperature cycling characteristics, and the like). This allows customized adjustment and control for viscosity, thermal conductivity, and heat melt/flow properties to allow precise performance matching to various applications or requirements.
- [0040] The film material A is composed of two elements, namely, a thermally conductive filler and a phase change material. The phase change material is a substance or mixture that undergoes a phase change at, or just below, a 55 selected operating temperature. The thermally conductive filler increases the thermal conductivity of the phase change material and is preferably selected from a variety of materials having a bulk thermal conductivity of between about 0.5 and 1000.0 Watts/meter-K as measured according to ASTM D1530. Examples of suitable conductive fillers include, but are not limited to, boron nitride, aluminum oxide, nickel powder, copper flakes, graphite powder, powdered diamond,

and the like. Preferably, the particle size of the filler, the particle size distribution, and filler loading (concentration in the film) are selected to maximize packing and thus produce the most efficient thermal conductance. Preferably, the particle size of the filler is between about 2 and 100 microns.

[0041] The phase change substance is a mixture of two or more compatible components or materials that undergoes a reversible solid-liquid phase change at the operating temperature of the heating device. The viscosity of the phase change substance at the melting temperature is low enough to completely wet-out the heat sink/power device interface but high enough to prevent exudation and loss of contact. The viscosity of the phase change substance at the operating temperature of the microprocessor or power supply assemblies (typically operating in a temperature range from 50°C to 100°C) is preferably between 1 and 100 poise and more preferably from 5-50 poise. More preferably, the phase change substance maintains a viscosity of between 5 and 50 poise over the temperature range of 60-120°C and has a melting point in the range of 30-120°C.

[0042] When cooled below its melting point, the phase change substance solidifies without a significant change in volume, thereby maintaining intimate contact between the heat sink 14 and the power device 12.

[0043] The first component of the phase change substance is a polymer component which includes a polymer. Suitable polymers include single or multi-component elastomers, consisting of one or more of the following: silicone, acrylic, natural rubber, synthetic rubber, or other appropriate elastomeric materials. Examples of such elastomers include styrene butadiene rubbers, both di-block and tri-block elastomers (e.g., Kraton® from Shell Chemicals), nitrile, natural rubber, polyester resins, combinations thereof, and the like, where the Mooney viscosity can range up to 40 ML4. Examples of suitable acrylic polymers include AeroSet 1085, AeroSet 414, AeroSet 1845, AeroSet 1081, and AeroSet 1452, obtainable from Ashland Chemicals.

[0044] The second component of the phase change substance is a melting point component. This component influences the melting point of the phase change substance for achieving a melting point of the film at around the operating temperature. Examples of suitable melting point components include C<sub>12</sub>-C<sub>16</sub> alcohols, acids, esters, and waxes, low molecular weight styrenes, methyl triphenyl silane materials, combinations thereof, and the like. Suitable C<sub>12</sub>-C<sub>12</sub>-C<sub>16</sub> acids and alcohols include myristyl alcohol, cetyl alcohol, stearyl alcohol, myristyl acid, and stearic acid. Preferred waxes include microcrystalline wax, paraffin waxes, and other wax-like compounds, such as cyclopentane, heceicosyl; 2-heptadecanone; pentacosanyl; silicic acid, tetraphenyl ester; octadecanoic acid; 2-[2-[2-(2hydroxyethoxy) ethoxy] ethyl ester; cyclohexane, docosyl; polystyrene; polyamide resins; disiloxane 1,1,1, trimethyl-3,3; and triphenyl silane.

[0045] The polymer component provides the phase change material with body (viscosity) to prevent the melting point component and filler from flowing out from between the heat sink and the microprocessor heat source. It thus acts as a viscosity controller. It also provides the film 10 with flexibility, handleability, and other film forming characteristics at ambient temperatures. Without the polymer component present, the melting point component would be brittle and tend to fracture and disintegrate at room temperature.

[0046] The melting point component melts at around the operating temperature and dissolves the polymer component in the melting point component. The viscosity of the liquefied melting point component drops as the polymer component dissolves in it. The resulting viscosity is sufficient for the material A to flow and wet the adjacent surfaces of the heat sink 14 and microprocessor heat source 12, creating thermal conduction paths and excellent thermal contact. Surface discontinuities in the heat sink and microprocessor heat source are filled with the material A. However, the viscosity is not so low that the material A flows out from between the parts 12, 14. To ensure that the thermal interface material has a relatively broad melting point, yet does not flow too readily at room temperatures, a combination of melting point components having differing melting points may be employed. For example, a combination of a C<sub>16</sub> component, such as cetyl alcohol, with a C<sub>14</sub> component, such as myristyl alcohol, yields a thermal interface material with good handling properties.

[0047] Preferably, the solubility parameter ( $\delta$ ) of the polymer component is within +1 and -1 of the solubility parameter(s) of the melting point, wax like component(s). This provides a level of compatibility between the liquefiable components.

[0048] Preferably, the material A comprises:

[0049] 1) from 10-80% polymer, more preferably 10-70%, most preferably from about 15 to about 50% polymer by weight;  
2) from 10-80% filler, more preferably 10-70%, most preferably, from about 15 to about 60% filler by weight; and  
3) from 10-80% melting point component, more preferably 15-70%, most preferably, from about 20% to about 60% melting point component by weight.

[0050] The material A may also contain other ingredients, such as colorants, e.g., for identifying the particular properties of the material; antioxidants, for improving storage properties; wetting agents, for improving contact with microprocessor components, and the like.

[0051] To prepare the films 10, the components of the phase change material (polymer component and melting

point component) are mixed with the filler. To improve the spreading characteristics of the material A, a processing aid, such as a solvent may be added to the mixture. Suitable solvents include low boiling aromatic and aliphatic compounds, such as toluene, benzene, xylene, heptane, mineral spirits, ketones, esters, alcohols, such as isopropyl alcohol, and mixtures thereof. A particularly preferred solvent is toluene, or a mixture of toluene and isopropyl alcohol. The isopropyl alcohol assists in dissolving the melting point component in the mixture.

[0051] The mixture is heated to about 50 °C to disperse the components and then dried to form the film 10 on one of the release liners 22. During this stage, the solvent evaporates. Optionally, a reinforcing layer 42 is then laminated to the thermal interface material. Alternatively, reinforcing materials, such as fibers, may be mixed with the thermal interface material prior to drying the film on the release liner.

[0052] One or more layers of adhesive are then optionally applied to the film 10 (or to the reinforcing layer) and a second release liner applied to the adhesive. Alternatively, the film is applied directly to a microprocessor power assembly or heat sink.

[0053] Suitable adhesives for the adhesive layer include Dow PSA adhesives 750D1 and 6574 and Ashland 414. The adhesive may be coated to a thickness of about 0.0002-0.0004 inches.

[0054] In another alternative embodiment, the adhesive is first applied to the second release liner. The release liner with the film and the release liner with the adhesive are then sandwiched together, optionally with application of heat, to bond the adhesive layer to the thermal interface material. A reinforcing material 42, such as glass cloth, may be laminated to the adhesive layer or to the thermal interface material, prior to sandwiching the adhesive layer and thermal interface material together.

[0055] The multi-layer strip thus formed may be supplied on a reel or sectioned into suitably sized pieces.

[0056] While not intending to limit the scope of the invention, the following examples provide suitable compositions for the thermal interface material A and the properties thereof.

## EXAMPLES

[0057] In the following examples, components were obtained, as follows:

### Melting Point components:

[0058] Myristyl alcohol (a C<sub>14</sub> alcohol), cetyl alcohol (a C<sub>16</sub> alcohol, sold under the tradename Lorol C<sub>16</sub>), stearyl alcohol, myristyl acid, and stearic acid were obtained from Aldrich Chemical or Henkel Chemicals.

[0059] Petroleum waxes were obtained from Bareco, including Bareco SC4095, Bareco SC5050, Bareco 5051, Bareco 6513, and Ultraflex.

[0060] Low melting aromatic hydrocarbons were obtained from Hercules, such as Piccolastic A-50, A-25, and A75.

### Polymer Components:

[0061] Several acrylic polymers were obtained from Ashland Chemical including Aeraset 1845, Aeraset 1085, Aeraset 1081, Aeraset 414, and Aeraset 1452.

[0062] Natural Rubber was obtained.

[0063] A di-block copolymer of styrene and butadiene was obtained from Shell Chemical.

[0064] Di-block copolymers of styrene and EP rubber were obtained from Shell, such as Kraton 1107, 1102, 1652.

[0065] Low melting point aromatic hydrocarbon resins were obtained from Hercules, such as Piccolite A50.

### Filler materials:

[0066] Boron Nitride was obtained from Advanced Ceramics. Alumina (Al<sub>2</sub>O<sub>3</sub>) was obtained from Alcoa Aluminum.

### Other Additives

[0067] Irganox 330, and Irganox 1010, both antioxidants, were obtained from Ciba Geigy.

[0068] A wetting agent, KR38S, was obtained from Kendrick.

[0069] Pigments were added in some examples for aesthetic purposes.

### EXAMPLE 1

[0070] To prepare a thermal interface material A, Aeraset 1845, myristyl acid, and boron nitride were combined in a mixture with toluene in the following amounts:

	Ingredient	Weight %	Purpose of the Mixture
5	AeroSet 1845	40	polymer component
	Boron Nitride Powder	20	filler material
	Myristyl Acid	20	melting point component
10	Toluene	20	solvent

[0071] This mixture was heated to 500 and mixed until homogeneous. The mixture was then dried at 100 C° for 5 minutes on a release liner to form a film 10. The dry thickness of the film (measured after removal of the release liner) was .00511 (0.127mm), as were the films of Examples 2-17 below.

#### EXAMPLE 2

[0072] To prepare a thermal interface material, AeroSet 414, myristyl acid, and boron nitride were combined in a mixture with toluene in the following amounts:

	Ingredient	Weight %	Purpose of the Mixture
25	AeroSet 414	40	polymer component
	Boron Nitride Powder	20	filler material
	Myristyl Acid	20	melting point component
30	Toluene	20	solvent

[0073] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

#### EXAMPLE 3

[0074] AeroSet 1085, myristyl acid, and boron nitride were combined in a mixture with toluene in the following amounts:

	Ingredient	Weight %	Purpose of the Mixture
40	AeroSet 1085	40	polymer component
	Boron Nitride Powder	20	filler material
	Myristyl Acid	20	melting point component
45	Toluene	20	solvent

[0075] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

#### EXAMPLE 4

55 [0076] AeroSet 1845, myristyl acid, and alumina were combined in a mixture with toluene in the following amounts:

5	Ingredient	Weight %	Purpose of the Mixture
10	Aeroset 1845	40	polymer component
	Alumina Powder	20	filler material
	Myristyl Acid	20	melting point component
	Toluene	20	solvent

[0077] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

#### 15 EXAMPLE 5

[0078] Aeroset 1845, cetyl alcohol, and boron nitride were combined in a mixture with toluene in the following amounts:

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Ingredient	Weight %	Purpose of the Mixture
Aeroset 1845	40	polymer component
Boron Nitride Powder	20	filler material
Cetyl alcohol	20	melting point component
Toluene	20	solvent

30

[0079] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

#### EXAMPLE 6

35 [0080] Aeroset 1845, myristyl alcohol, and boron nitride were combined in a mixture with toluene in the following amounts:

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Ingredient	Weight %	Purpose of the Mixture
Aeroset 1845	40	polymer component
Boron Nitride Powder	20	filler material
Myristyl alcohol	20	melting point component
Toluene	20	solvent

50

[0081] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

55

#### EXAMPLE 7

[0082] Aeroset 1845, stearyl alcohol, and boron nitride were combined in a mixture with toluene in the following amounts:

	Ingredient	Weight %	Purpose of the Mixture
5	Aeroset 1845	40	polymer component
	Boron Nitride Powder	20	filler material
	Stearyl alcohol	20	melting point component
10	Toluene	20	solvent

[0083] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

#### 15 EXAMPLE 8

[0084] Aeroset 1081, myristyl acid, and boron nitride were combined in a mixture with toluene at the ratios:

	Ingredient	Weight %	Purpose of the Mixture
20	Aeroset 1081	40	polymer component
	Boron Nitride Powder	20	filler material
25	Myristyl Acid	20	melting point component
	Toluene	20	solvent

30 [0085] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

#### EXAMPLE 9

[0086] Natural rubber, Bareco SC5050, Piccolastic A50, and boron nitride powder were combined with toluene in  
35 the following amounts:

	Ingredient	Weight%	Purpose of the Mixture
40	Natural rubber	10	polymer component
	Boron Nitride Powder	30	filler material
	Bareco SC 5050	20	melting point component
45	Toluene	30	solvent
	Piccolastic A50	10	melting point component

50 [0087] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

#### EXAMPLE 10

[0088] Natural rubber, Bareco SC5050, Piccolastic A50, and boron nitride powder were combined with toluene as  
follows:

55

5	Ingredient	Weight %	Purpose of the Mixture
	Natural rubber	20	polymer component
10	Boron Nitride Powder	10	filler material
	Bareco SC5050	20	melting point component
	Toluene	30	solvent
	Piccolastic A50	20	melting point component

[0089] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

15 EXAMPLE 11

[0090] Natural rubber, Bareco SC5050, Piccolastic A25, and boron nitride powder were combined with toluene, as follows:

20	Ingredient	Weight %	Purpose of the Mixture
25	Natural rubber	10	polymer component
	Boron Nitride Powder	30	filler material
	Bareco SCS050	20	melting point component
30	Toluene	30	solvent
	Piccolastic A25	10	melting point component

[0091] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

35 EXAMPLE 12

[0092] Natural rubber, Bareco SC5050, and boron nitride powder were combined with toluene as follows:

40	Ingredient	Weight %	Purpose of the Mixture
	Natural rubber	20	polymer component
45	Boron Nitride Powder	40	filler material
	Bareco SC5050	10	melting point component
	Toluene	30	solvent

50 [0093] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

EXAMPLE 13

[0094] Kraton 1107, Bareco SC5050, Piccolastic A50, and Boron Nitride were combined with toluene as follows:

5	Ingredient	Weight %	Purpose of the Mixture
	Kraton 1107	10	polymer component
	Boron Nitride Powder	30	filler material
10	Bareco SC5050	20	melting point component
	Toluene	30	solvent
	Piccolastic A50	10	melting point component

[0095] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

15 EXAMPLE 14

[0096] Kraton 1102, Bareco SC5050, Piccolastic A50, and boron nitride were combined as follows:

20	Ingredient	Weight %	Purpose of the Mixture
	Kraton 1102	10	polymer component
25	Boron Nitride Powder	30	filler material
	Bareco SC5050		melting point component
	Toluene	30	solvent
30	Piccolastic A50	10	melting point component

[0097] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

EXAMPLE 15

[0098] Kraton 1652, Bareco SC5050, Piccolastic A50, and boron nitride were combined with toluene as follows:

40	Ingredient	Weight %	Purpose of the Mixture
	Kraton 1652	10	polymer component
	Boron Nitride Powder	30	filler material
45	Bareco SC5050	20	melting point component
	Toluene	30	solvent
	Piccolastic A50	10	melting point component

50 [0099] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

EXAMPLE 16

[0100] Kraton 1107, Bareco SC5050, and Boron Nitride were combined with toluene as follows:

Ingredient	Weight %	Purpose of the Mixture
Kraton 1107	10	polymer component
Boron Nitride Powder	30	filler material
Bareco SC5050	20	melting point component
Toluene	30	solvent

[0101] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

#### EXAMPLE 17

[0102] Natural Rubber, Bareco SC5050, and alumina powder were combined with toluene as follows:

Ingredient	Weight %	Purpose of the Mixture
Natural rubber	10	polymer component
Alumina	30	filler material
Bareco SC5050	20	melting point component
Toluene	40	solvent

[0103] This mixture was heated, coated and dried on a release liner, using the method of Example 1.

#### EXAMPLE 18

[0104] Ashland adhesive, AeroSet 1081, Lorol C<sub>16</sub> (cetyl alcohol), isopropyl alcohol, boron nitride, Irganox 330 (an antioxidant, obtained from Ciba Geigy), Irganox 1010 (an antioxidant, obtained from Ciba Geigy), KR38S (a wetting agent, obtained from Kendrich), and pigments (for aesthetic purposes), were combined in a mixture as listed below with toluene at the following ratios:

Ingredient	Weight Percent
Irganox 330	0.32%
Boron Nitride	23.76%
Isopropyl Alcohol	22.45%
Irganox 1010	0.32%
KR 38S	0.24%
Toluene	4.82%
Pigment Red (UDC Red)	0.08%
Pigment Yellow (UDC yellow)	0.48%
Cetyl Alcohol	17.11%
Cetyl Alcohol	9.51%
AeroSet Adhesive 1081 Adhesive 1081	20.91

[0105] This mixture was heated to 50°C and mixed until a homogeneous solution developed. The cetyl alcohol was added in two stages, although the total amount may also be added at one time. The solution was then coated onto a paper previously coated with a silicone release film. The coating was dried at 100°C for 5 minutes. The dry thickness was .003" thick. The thermal interface material thus formed was removed from the liner and tested in an Anter "Unitherm™" thermal test apparatus, model 2021 SX67 according to ASTM D1530. The thermal resistance of the sample was 0.03 °C-in<sup>2</sup>/W. Thermal conductivity was 1.2 W-M/K.

#### EXAMPLE 19

[0106] Aeroset 1081, Lorol C16 alcohol, and boron nitride were combined in a mixture with other ingredients and toluene at the following ratios:

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Component	Percent of total
Aeroset 1081	20.973
Isopropyl alcohol	22.523
Toluene	4.493
Boron Nitride	23.84
Cetyl Alcohol	26.697
Irganox 330	0.323
Irganox 1010	0.323
KR38S	0.27
UCD Yellow	0.476
UCD Red	0.081

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[0107] The mixture was heated to 50°C and mixed until homogeneous. This solution was then coated onto a liner paper coated with silicone release. This was dried at 100°C for 5 minutes. The dry thickness was 0.003" thick. The thermal interface material was removed from the liner and tested in an Anter "Unitherm™" thermal test apparatus, model 2021 SX67 according to ASTM D1530. The thermal resistance of the sample was 0.03°C-in<sup>2</sup>/W. Thermal conductivity was 1.2 W-M/K.

#### EXAMPLE 20

[0108] An adhesive (Dow PSA 750D1) was coated onto a liner sheet (Furon product number 9022) at 0.0003" thick. The PSA coated liner was then laminated to a sheet of woven fiberglass as a reinforcing material (Product #106 from BGF industries). A liner sheet with a thermal interface material was prepared according to the method of Example 19. Then the polymeric coated sheet of Example 19 and the fiberglass laminated with the PSA coated liner were heated and laminated together at 130°F to provide a multiple layer strip in the following order:

45

Furon liner sheet,  
PSA adhesive,  
fiberglass sheet,  
thermal interface material, and  
release-coated liner.

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[0109] The thermal interface material laminated to glass cloth (fiberglass) was removed from the two liner sheets and tested in an Anter "Unitherm™" thermal test apparatus, model 2021 SX67 according to ASTM D1530. The thermal resistance of the sample was 0.04 °C-in<sup>2</sup>/W. Thermal conductivity was 1.1 W-M/K.

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#### EXAMPLE 21

[0110] An adhesive (Dow PSA 750D1) was coated onto a liner (Furon product number 9022) at 0.0003" thick. The

PSA coated sheet was then laminated to fiberglass (fiberglass #106 from BGF industries). A liner sheet coated with thermal interface material was prepared according to the method of Example 19. Then, the polymeric coated sheet of Example 19 and fiberglass laminated with the PSA coated liner were heated and laminated together at 130 °F. The thermal interface material laminated to glass cloth was removed from the liners and tested in an Anter "Unitherm™" thermal test apparatus, model 2021 SX67 according to ASTM D1530. The thermal resistance of the sample was 0.04 °C-in<sup>2</sup>/W. Thermal conductivity was 1.1 W-M/K.

#### EXAMPLE 22

[0111] An adhesive (Dow PSA 6574) was coated onto a liner (Furon product number 9022) at 0.0003" thick. The PSA coated sheet was then laminated to fiberglass (fiberglass #106 from BGF industries). A liner sheet coated with thermal interface material was prepared according to the method of Example 19. Then, the polymeric coated sheet of Example 19 and fiberglass laminated with the PSA coated liner were heated and laminated together at 130 °F. The thermal interface material laminated to glass cloth was removed from the liners and tested in an Anter "Unitherm™" thermal test apparatus, model 2021 SX67 according to ASTM D1530. The thermal resistance of the sample was 0.04 °C-in<sup>2</sup>/W. Thermal conductivity was 1.1 W-M/K.

#### EXAMPLE 23

[0112] An adhesive (Dow PSA 6574) was coated onto a liner (Furon product number 750D1) at 0.0003" thick. The PSA coated sheet was then laminated to 0.002" thick aluminum foil. A liner sheet with thermal interface material was prepared according to the method of Example 19. Then, the polymeric coated sheet of Example 19 and aluminum foil laminated with the PSA coated liner were heated and laminated together at 130 °F. The thermal interface material laminated to glass cloth was removed from the liners and tested in an Anter "Unitherm™" thermal test apparatus, model 2021 SX67 according to ASTM D1530. The thermal resistance of the sample was 0.025 °C-in<sup>2</sup>/W. Thermal conductivity was 1.5 W-M/K.

#### EXAMPLE 24

[0113] An adhesive (Ashland 414) was coated onto a liner (Furon liner product number 1018) at 0.0002" thick. The adhesive coated liner was then laminated to fiberglass (fiberglass #106 from BGF industries). A liner sheet coated with thermal interface material was prepared according to the method of Example 19. Then, the polymeric coated sheet of Example 19 and fiberglass laminated with the PSA coated liner were heated and laminated together at 130 °F. The thermal interface material laminated to glass cloth was removed from the liners and tested in an Anter "Unitherm™" thermal test apparatus, model 2021 SX67 according to ASTM D1530. The thermal resistance of the sample was 0.04 °C-in<sup>2</sup>/W. Thermal conductivity was 1.1 W-M/K.

#### EXAMPLE 25

[0114] The films produced in examples 1-17 were tested for chemical and physical properties. Table 1 summarizes the properties for each of the examples. As can be seen from Table 1, the melting point of the film can be adjusted by selecting the components of the melting component. Thermal impedance and thermal conductivity were measured on liner free samples of the film using Anter "Unitherm™" thermal test apparatus, model 2021 SX67, according to ASTM D1530. Differential Scanning Calorimetry (DSC) was used to determine the melting point of the compound.

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Example	Melting Point DSC °C	Thermal Impedance (°C in <sup>2</sup> /W)	Thermal Conductivity (W-m/K)	Thermal Resistance Pentium Pro (°C/W)	DMA Viscosity @ 100°C (Pa.S)	Solubility Parameter Wax $\delta$	Solubility Parameter Polymer $\delta$
1	58	.07	0.8	0.30		10.1	10.5
2	58	.08	0.8	0.28	50	10.1	10.5
3	58	.07	0.8	0.22		10.1	10.5
4	58	.07	0.7	0.29		10.1	10.5

(continued)

Example	Melting Point DSC°C	Thermal Impedance (°C in <sup>2</sup> /W)	Thermal Conductivity (W-m/K)	Thermal Resistance Pentium Pro (°C/W)	DMA Viscosity @ 100°C (Pa.S)	Solubility Parameter Waxδ	Solubility Parameter Polymer δ
5	50	.07	0.8	0.21	15	10.7	10.5
6	40	.07	0.8	0.19	10	10.5	10.5
7	60	.07	0.8	0.27		10.5	10.5
8	58	.07	0.8	0.30		10.4	10.5
9	45	.07	0.8	0.35		7.4	7.4
10	45	.10	0.5	0.33		7.4	7.4
11	45	.07	0.8	0.33		7.4	7.4
12	45	.07	1.4	0.34		7.4	7.4
13	47	.07	0.8	0.30		7.4	7.4
14	47	.07	0.8	0.28		7.4	7.4
15	47	.07	0.8	0.27		7.4	7.4
16	45	.07	0.8	0.29		7.4	7.4
17	45	.07	0.8	0.35		7.4	7.4

**EXAMPLE 26**

[0115] Mounting pressure for various films was calculated using the following formula:

$$P = \frac{T \times N}{0.2 \times D \times A}$$

where

N = Number of fasteners

D = Fastener diameter (inches)

A = Surface contact area (sq. in.)

[0116] FIGURE 7 shows a plot of thermal impedance versus the mounting pressure for three different materials. The first two are a silicone coated fabric using Bergquist SP 400, a silicone coated polyimide (Kapton®), prepared according to U.S. Patent No. 4,574,879 and the third is the film of Example 6 of this application. As can be seen from FIGURE 7, the film of Example 6 has low thermal impedance over the entire range of mounting pressures.

[0117] The invention has been described with reference to the preferred embodiments. It should be apparent that modifications and alterations will occur to others upon a reading and understanding of the preceding specification. It is intended that the invention be construed as including all such alterations and modifications insofar as they come within the scope of the appended claims or the equivalents thereof.

Claims

1. A thermal interface material which undergoes a phase change at microprocessor operating temperatures to transfer heat generated by a heat source to a heat sink, the material comprising:

a phase change substance which softens at about the operating temperature of the heat source, the phase change substance including:

a polymer component, and

a melting component mixed with the polymer component, which modifies the temperature at which the phase change substance softens; and

a thermally conductive filler dispersed within the phase change substance.

- 5        2. The thermal interface material according to claim 1, wherein the phase change substance has a viscosity of from 1 to 100 poise at the operating temperature of the heat source.
- 10      3. The thermal interface material according to claim 1 or 2, wherein the phase change substance has a viscosity of from 5 to 50 poise in the temperature range of 60 to 120°C.
- 15      4. The thermal interface material according to claim 1, 2 or 3, wherein the phase change substance has a melting point of 30-120 °C.
- 20      5. The thermal interface material according to any preceding claim, wherein the polymer component includes an elastomer selected from the group consisting of silicone, acrylic polymers, natural rubber, synthetic rubber, and combinations thereof.
- 25      6. The thermal interface material according to any preceding claim, wherein the polymer component has a Mooney viscosity of up to 40 ML4.
- 30      7. The thermal interface material according to any preceding claim, wherein the melting point component is selected from the group consisting of C<sub>12</sub>-C<sub>16</sub> alcohols, acids, esters, petroleum waxes, wax-like compounds, low molecular weight styrenes, methyl triphenyl silane materials, and combinations thereof.
- 35      8. The thermal interface material according to claim 7, wherein the melting point component is a C<sub>12</sub>-C<sub>16</sub> alcohol or acid selected from the group consisting of myristyl alcohol, cetyl alcohol, stearyl alcohol, myristyl acid, stearic acid, and combinations thereof.
- 40      9. The thermal interface material according to claim 7, wherein the melting point component is a wax or a waxlike compound selected from the group consisting of microcrystalline wax, paraffin waxes, cyclopentane, heceicosyl, 2-heptadecanone, pentacosanyl, silicic acid, tetraphenyl ester, octadecanoic acid, 2-[2-[2-(2hydroxyethoxy)ethoxy]ethoxy]ethyl ester, cyclohexane docosyl, polystyrene, polyamide resins, disiloxane 1,1,1, trimethyl-3,3, triphenyl silane, and combinations thereof.
- 45      10. The thermal interface material according to any preceding claim, wherein the polymer component has a solubility parameter which is within +1 and -1 of the solubility parameter of the melting point component.
- 50      11. The thermal interface material according to claim 1, comprising from 10-80% by weight of the polymer component; from 10-80% by weight of the filler; and from 10-80% by weight of the melting point component.
- 55      12. The thermal interface material according to claim 11, comprising from 10-70% by weight of the polymer component; from 10-70% by weight of the filler; and from 15-70% by weight of the melting point component.
- 60      13. The thermal interface material according to any preceding claim, wherein the thermally conductive filler has a bulk thermal conductivity of between about 0.5 and 1000 watts meter per degree Kelvin.
- 65      14. The thermal interface material according to any preceding claim, wherein the thermal interface material has a thermal conductivity of at least 0.8 watts meter per degree Kelvin.
- 70      15. The thermal interface material according to any preceding claim, wherein the thermally conductive filler is selected from the group consisting of boron nitride, aluminum oxide, nickel powder, copper flakes, graphite powder, powdered diamond, and combinations thereof.
- 75      16. The thermal interface material according to any preceding claim, wherein the thermally conductive filler has an average particle size of from about 2 to 100 microns.
- 80      17. A multi-layer strip comprising:

a first layer of a thermal interface material as defined in any of claims 1 to 16,  
and

a second layer disposed on a side of the thermal interface material, the second layer including at least one of:

- 5            a protective releasable liner, and  
              a layer of an adhesive material.

18. The multi-layer strip according to claim 17, wherein the second layer includes a protective releasable liner and the strip further comprises:

- 10            a layer of an adhesive material disposed on a second side of the thermal interface material.

19. The multi-layer strip according to claim 18, further including:

- 15            a second protective release liner disposed on the layer adhesive material.

20. The multi-layer strip according to any of claims 17 to 19, further including a reinforcing material in contact with the interface material.

- 20            21. The multi-layer strip according to claim 20, wherein the reinforcing material is selected from the group consisting of fiberglass and aluminum foil.

22. The multi-layer strip according to any of claims 17 to 21, wherein the protective liner includes a substrate coated with a release coating.

- 25            23. A method of providing a thermal interface between a heat source and a heat sink, the method comprising:

30            interposing a thermal interface material as defined in any of claims 1 to 16 between the heat source and heat sink which material softens at about the operating temperature of the heat source to provide a thermal interface between the heat source and the heat sink during operation of the heat source.

24. The method according to claim 23, further including:

- 35            adhering the thermal interface material to one of the heat source and the heat sink by application of heat.

25. The method according to claim 23 or 24, further including:

40            adhering the thermal interface material to one of the heat source and the heat sink with a layer of an adhesive material.

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FIG. 1A  
FIG. 1

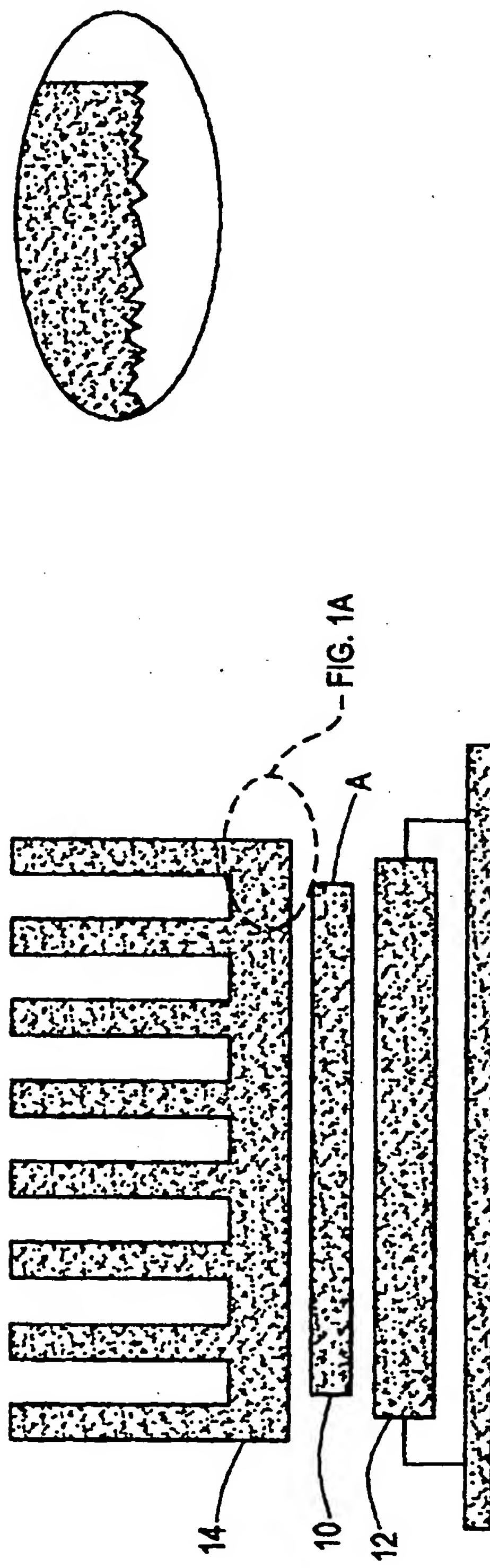


FIG. 2

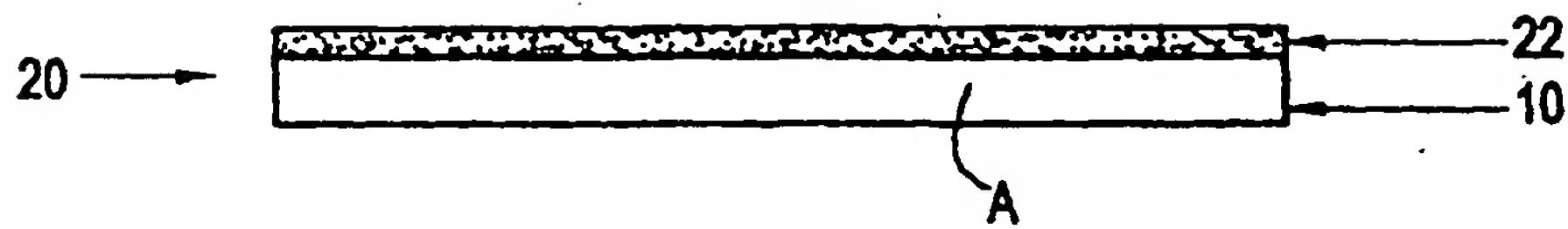


FIG. 3

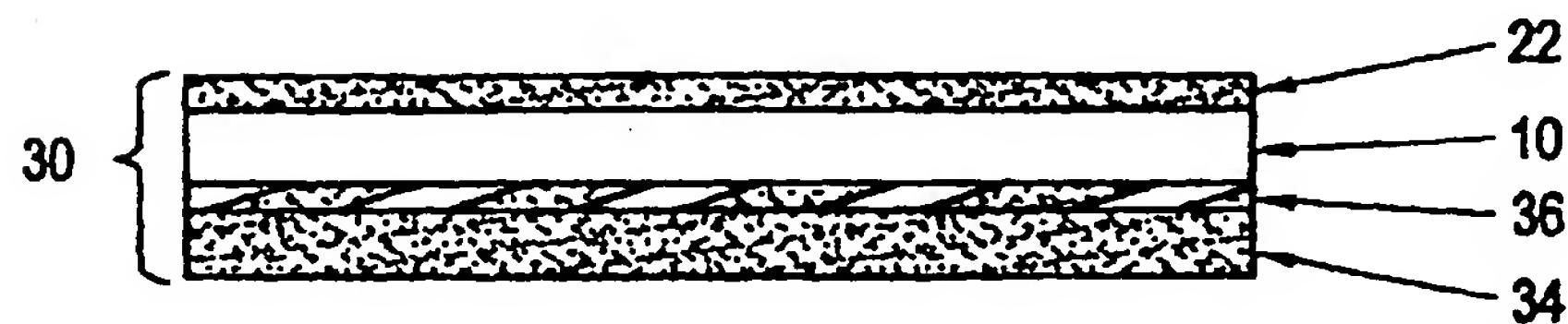


FIG. 4

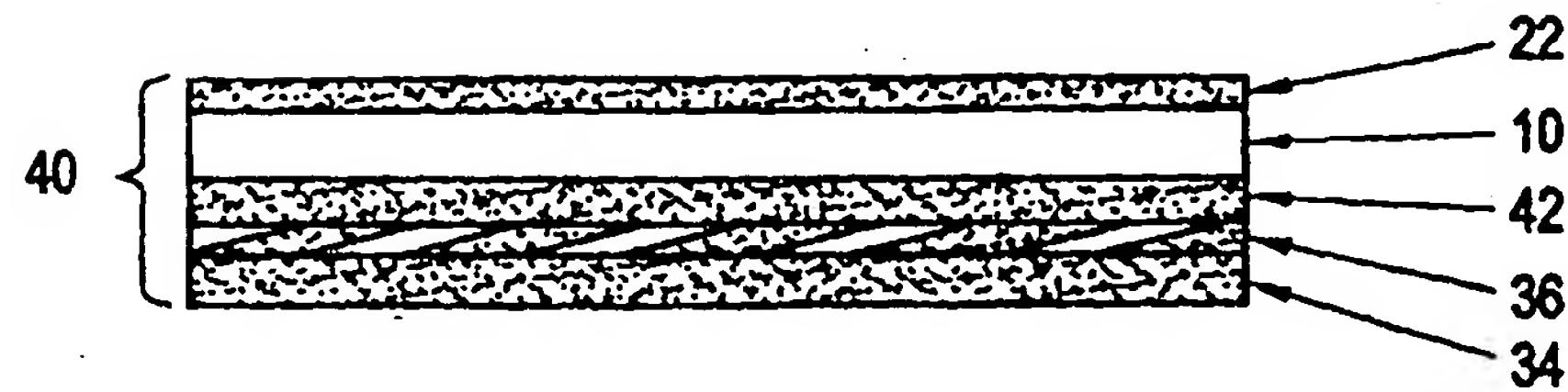


FIG. 5

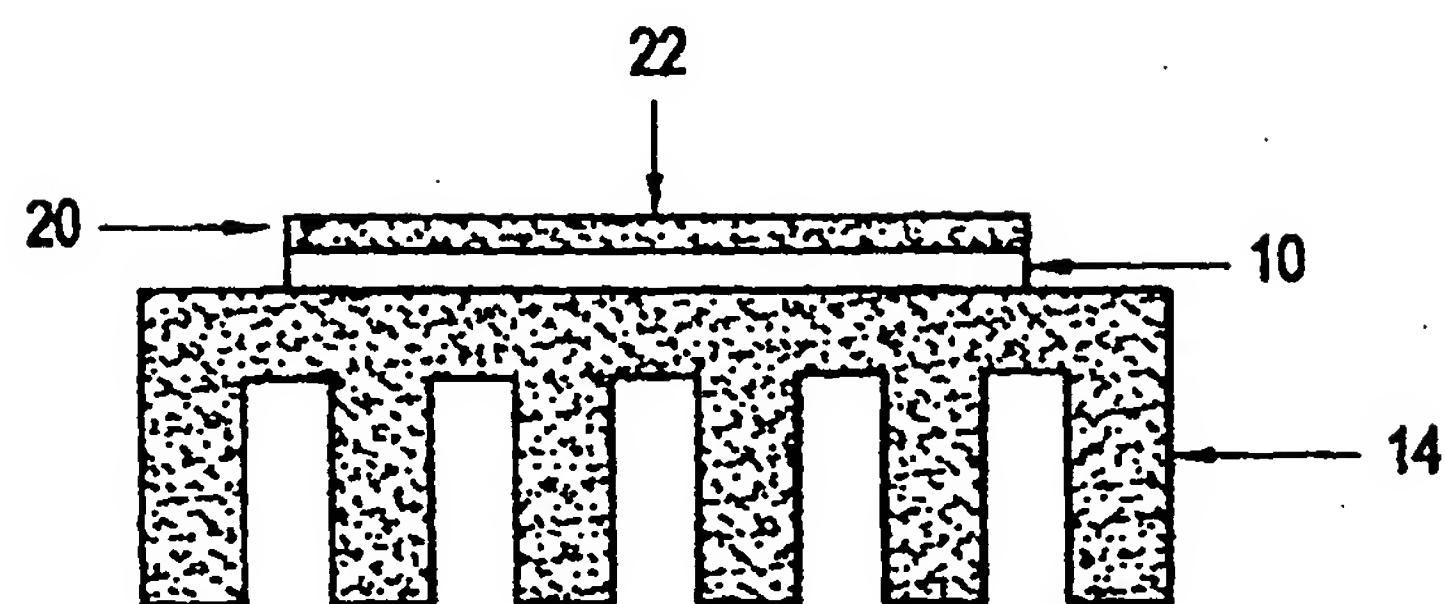


FIG. 6

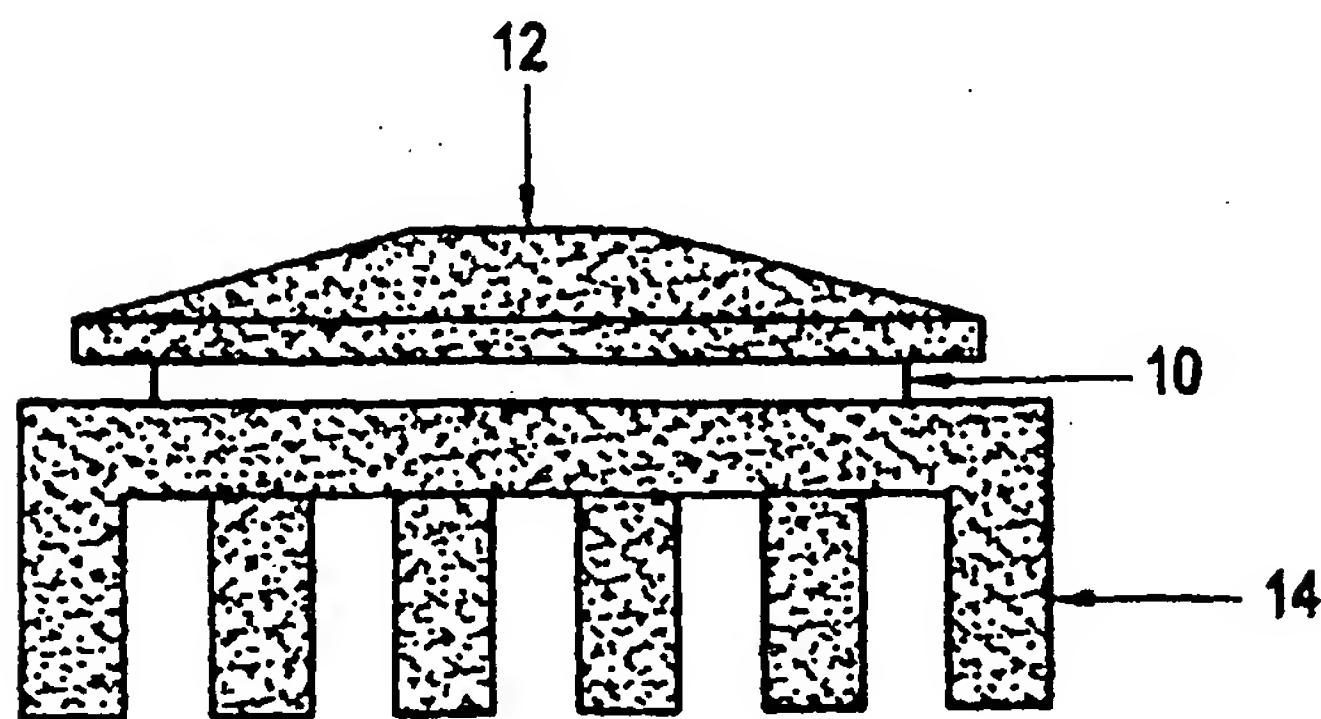
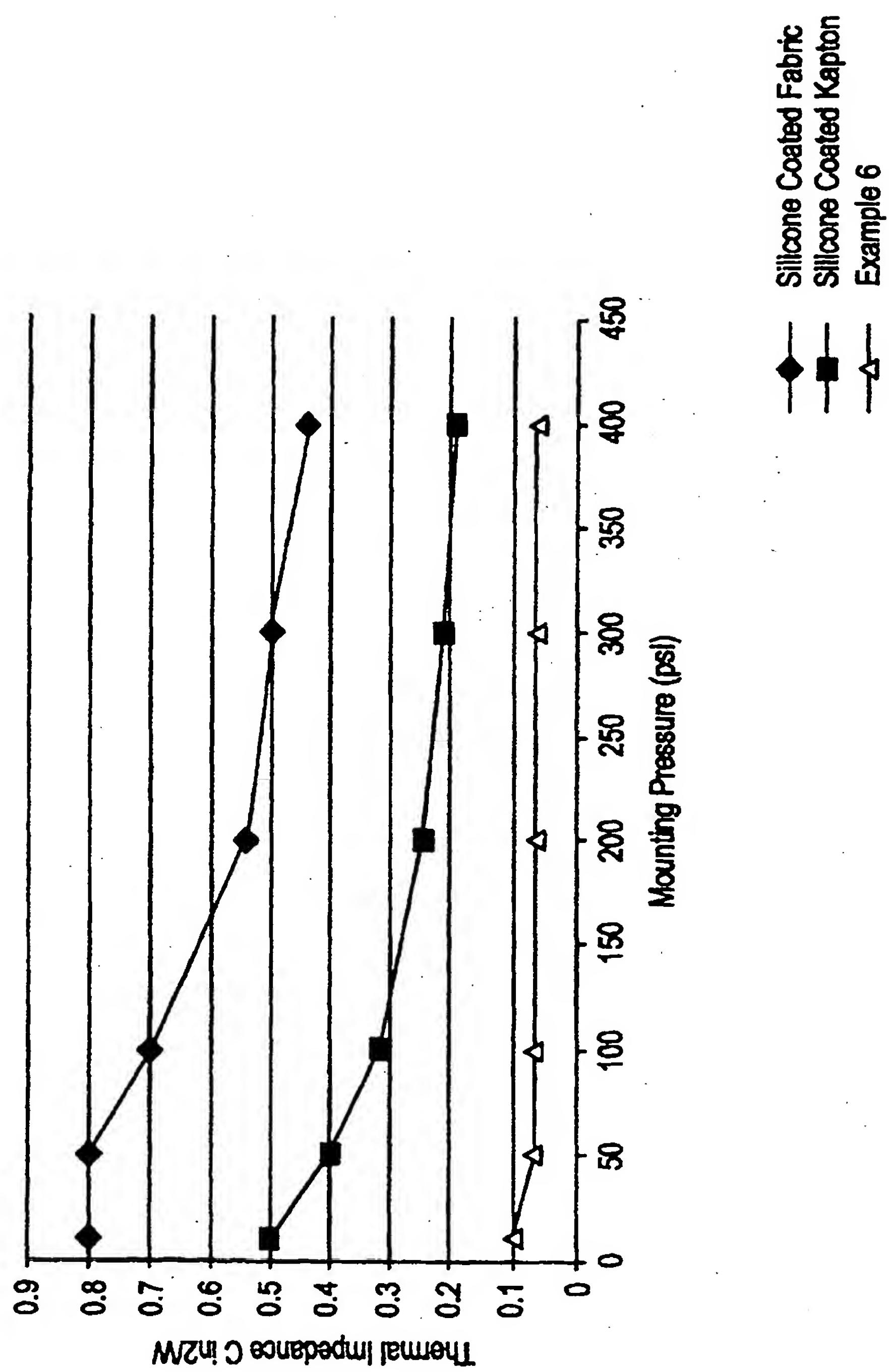


FIG. 7





European Patent  
Office

## EUROPEAN SEARCH REPORT

Application Number  
EP 00 30 5747

DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int.Cl.)						
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim							
X	WO 99 19909 A (GORE ENTERPRISE HOLDINGS INC) 22 April 1999 (1999-04-22) * the whole document * ---	1,4,7,9, 11-16,23	C09K5/06 H01L23/373						
X	WO 97 41599 A (PARKER HANNIFIN CORP) 6 November 1997 (1997-11-06) * the whole document * ---	1,4,5, 7-9,13, 15-17,23							
A	EP 0 813 244 A (BERGQUIST CO) 17 December 1997 (1997-12-17) * page 3, line 5 - page 4, line 14 * * page 7, line 45 - page 8, line 14 * * claims 1,2; figures * ----	1,3,15, 16							
A	US 5 679 457 A (BERGERSON STEVEN E) 21 October 1997 (1997-10-21) * claims * -----	1,5,13, 15-17,23							
			TECHNICAL FIELDS SEARCHED (Int.Cl.)						
			C09K H01L						
<p>The present search report has been drawn up for all claims</p> <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 33%;">Place of search</td> <td style="width: 33%;">Date of completion of the search</td> <td style="width: 33%;">Examiner</td> </tr> <tr> <td>THE HAGUE</td> <td>11 October 2000</td> <td>Puetz, C</td> </tr> </table> <p><b>CATEGORY OF CITED DOCUMENTS</b></p> <p>X : particularly relevant if taken alone  Y : particularly relevant if combined with another document of the same category  A : technological background  O : non-written disclosure  P : intermediate document</p> <p>T : theory or principle underlying the invention  E : earlier patent document, but published on, or after the filing date  D : document cited in the application  L : document cited for other reasons  &amp; : member of the same patent family, corresponding document</p>				Place of search	Date of completion of the search	Examiner	THE HAGUE	11 October 2000	Puetz, C
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THE HAGUE	11 October 2000	Puetz, C							

**ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.**

EP 00 30 5747

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11-10-2000

Patent document cited in search report		Publication date		Patent family member(s)		Publication date
WO 9919909	A	22-04-1999		US 5945217 A		31-08-1999
				AU 9373598 A		03-05-1999
WO 9741599	A	06-11-1997		AU 723258 B		24-08-2000
				AU 1807797 A		19-11-1997
				EP 0956590 A		17-11-1999
				JP 2000509209 T		18-07-2000
EP 0813244	A	17-12-1997		US 5950066 A		07-09-1999
				CA 2207114 A		14-12-1997
				JP 10067910 A		10-03-1998
US 5679457	A	21-10-1997		NONE		